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COMPLETE SPECIFICATION

Detergent Compositions

We, GENERAL ANILINE & FILM CORPORATION, a corporation organized and existing under the laws of the State of Delaware, United States of America, of
5 230, Park Avenue, New York, County and State of New York, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by
10 which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to an improved germicidal composition particularly
15 adapted for use as a germicidal detergent, comprising a solution of iodine in a water soluble, nonionic surface active agent having a polyglycol ether group.

We have found that the water soluble,
20 nonionic surface active agents which contain a polyglycol ether group readily dissolve substantial amounts of iodine. These nonionic surface active agents of themselves have no bactericidal or
25 bacteriostatic effect and while the iodine is known to have powerful germicidal effect, the amount which has been required has been so great as to restrict its economical use in detergent sanitizers. In
30 addition, iodine is only sparingly soluble in water, so that it does not readily lend itself to use in aqueous media. We have now found that water soluble, nonionic surface active agents containing poly-
35 glycol ether groups in which there has been dissolved iodine, exert substantially greater germicidal effects than other compositions containing a corresponding amount of iodine. In addition, these
40 solutions may be diluted with water without precipitation of iodine in contrast with solutions of iodine in other solvents such as alcohol from which the iodine precipitates on dilution with water. The
45 novel solution of the present invention is therefore not only an efficient germicidal

composition, but one which can be recommended from an economical standpoint.

We have found that solutions of elemental iodine in these nonionic surface-
50 active agents are particularly desirable in that a stable product is obtained which is effective in the dilute solutions normally used for cleansing operations particularly in food handling plants and institu-
55 tional cleaning. The iodine is readily incorporated in these nonionic surface-active agents by merely adding the iodine to these nonionic surface-active agents. It has been found that ordinary iodine
60 crystals will dissolve by merely being allowed to stand in the nonionic surface-active agent. However, solution may be expedited by grinding and stirring the
65 iodine in the nonionic surface-active agent. Up to 15% of iodine has been readily added to the nonionic surface-active agent and larger percentages
70 apparently can be added but are not necessary. The solution was found to be stable on storage. The amount of iodine to be added to the nonionic surface-active agent will depend on the intended use of
75 the product since obviously with products intended for use at high dilutions more iodine must be present in order to have effective bactericidal effect than in products intended for use at lower dilutions. However, the amount of iodine required
80 in the final solution used in any given cleaning operation when employing solutions of this invention is substantially less than the amount of iodine required alone to effect the same bactericidal result.
85 Where more rapid disinfection is required the amount of iodine in the solution might be increased but would still be less than the quantity of iodine required alone to accomplish the same
90 effect.

It has been found that a water soluble nonionic surface active agent, having a

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10 active agent having a polyglycol ether group which was produced by condensing a nonyl phenol with about 8 molar proportions of ethyleneoxide had dissolved in it about 5% by weight of iodine. The thus obtained solution was tested against *Staph. aureus* in the usual phenol coefficient tests. In these tests it was found that this solution, when diluted with water so that there was present about 20 part of iodine in 60,000 parts of material under test (or roughly 1 part of nonionic surface active agent in 3,000 parts of solution under test) effected complete kill in 5 minutes, while at dilutions of 25 1:80,000 iodine complete kill was effected in 10 minutes. As compared with this in the same test, using Lugol's solution, complete kill was effected in 5 minutes at iodine dilution of 1:25,000 and in 10 30 minutes at a dilution of 1:40,000.

As indicated above, up to 15% of iodine can readily be dissolved in the nonionic surface-active agents and the exact amount which should be dissolved 35 depends on the germicidal activity which is desired and on the concentration at which the solution is to be used. For a general purpose detergent for household and similar use, from 1 to 3% of iodine based on the amount of nonionic surface-active agent produces a solution having effective bactericidal action when used in dilutions of the order of 1:3000.

Not only does the solution of nonionic 45 surface-active agent and iodine have a synergistic bactericidal effect but it has been found that the vapor pressure of the iodine in the solution is reduced to substantially zero since the solution has no odor of iodine and on heating the solution the starch test is negative. In addition it was found that the skin irritation and sensitization effects of the iodine were eliminated from the solution. Skin 55 patch tests on known iodine sensitive individuals were negative with the solution, whereas they showed 4+ and higher reactions to Lugol's solution. The solution can be used in contact with white cotton 60 materials which contain starch without any discoloration of these fabrics even in dilutions ad infinitum. Yet the quantity of iodine can be determined volumetrically on titration with sodium thiosulfate.

70 by condensing alkylene oxides with water-insoluble organic compounds containing at least six carbon atoms and having an active hydrogen, such as organic hydroxy compounds, alcohols, 80 primary and secondary amines, carbonylic acids, sulfonic acids and their amides. Compounds of this type are well known in the art and are disclosed along with suitable methods for their preparation, in U.S. Patents Nos. 1,970,578 and 85 2,213,476. They may be represented by the following general formula:



wherein R represents the residue of an organic compound containing at least 6 90 carbon atoms and an active hydrogen atom, R' represents hydrogen or lower alkyl and n represents an integer of from 3 to 100 or higher, and usually from 6 to 50. These compounds may readily be 95 obtained as disclosed in the above mentioned patents by condensing a polyglycol ether containing the required number of alkenoxy groups or an alkylene oxide, usually ethylene oxide (although 100 propylene or butylene oxides may be employed if desired) with a water-insoluble organic compound containing at least 6 carbon atoms and having an active hydrogen. The amount of alkylene oxide 105 condensed with the water insoluble organic compound having an active hydrogen, i.e. the length of the polyglycol ether chain, will depend primarily on the particular compound with which 110 it is condensed. As a convenient rule of thumb, approximately 1 mol of alkylene oxide should be employed for each two carbon atoms in the water insoluble organic compound containing an active 115 hydrogen with which it is condensed. However, the optimum amount of alkylene oxide can readily be determined in any particular case by preliminary test. 120

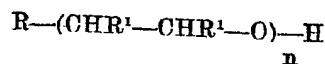
The liquid, nonionic polyglycol ether type surface-active agents derived from alkyl phenolic compounds have been found to be particularly valuable in detergent compositions, and, therefore, 125

the poly-alkylene oxide derivatives of such phenolic compounds are a preferred type of nonionic surface-active agents which are employed in the solutions of the present invention. Numerous compounds of this type, i.e. poly-alkylene oxide derivatives of phenolic compounds containing one or more alkyl substituents, which may be employed in the compositions of the present invention are described in U.S. Patent No. 2,213,477, and it is preferable to employ the water-soluble poly-alkylene oxide derivatives of alkyl phenolic compounds in which the total number of alkyl carbon atoms is between 6 and 18. As examples of such phenolic compounds may be mentioned the isomeric dibutyl and diamyl phenols and cresols, tripropyl phenol and cresol, secondary or tertiary isomeric heptyl, octyl, nonyl, decyl, undecyl, dodecyl, tetradecyl, cetyl, oleyl, and octadecyl phenols and cresols. Of particular value are the poly-alkylene oxide derivatives of secondary and tertiary alkyl substituted phenols and cresols obtained by condensing olefines of the type obtained in petroleum refining with phenols or cresols. In the case of products obtained by condensing phenol or cresol with olefines of from 3 to 5 carbon atoms such as propylene, butylene and amylene, it is desirable to employ the di-alkylated phenols or cresols, while in the case of compounds obtained by condensing a phenol or cresol with an olefine containing 8 or more carbon atoms, the mono-substituted derivatives are preferred. Particularly desirable derivatives can be obtained from the phenols and cresols containing a substituent derived from olefines containing from 8 to 18 carbon atoms, such as di-isobutylene and other alkylenes such as nonylene, decylene, undecylene, dodecylene, pentadecylene, octadecylene and mixtures thereof, and the dimers and trimers obtained by polymerization of such low molecular weight olefines as propylene, butylene, amylene or mixtures thereof may be advantageously used. However, the water-soluble poly-alkylene oxide derivatives of other water-insoluble organic compounds containing at least 6 carbon atoms and having an active hydrogen may be employed in the solutions of the present invention. Thus, the poly-alkylene oxide derivatives, described in U.S. Patent No. 1,970,578, of aliphatic organic hydroxy compounds, carboxy compounds and amino compounds may be employed if desired. As examples of water-insoluble higher fatty acids whose poly-alkylene oxide derivatives may be employed may be mentioned lauric, oleic, ricinoleic,

myristic and stearic acid or mixtures thereof, such as the mixtures obtained from animal and vegetable fats and oils or by the oxidation of such petroleum fractions as paraffin wax. There may also be employed poly-alkylene oxide derivatives of water insoluble aliphatic hydroxy compounds such as higher aliphatic alcohols, i.e., the alcohols corresponding to the fatty acids specified immediately above, particularly the alcohols obtainable by hydrogenation of the fatty acids or glycerides present in animal or vegetable oils and waxes such as coconut oil, and castor oil. There may also be used poly-alkylene oxide derivatives of higher molecular weight amines such as octyl amine, cetyl amine, oleyl amine, naphthyl amines, and alkyl anilines; also the polyalkylene oxide derivatives of organic mercapto compounds such as the products described in U.S. Patent No. 2,205,021, i.e. the polyalkylene oxide derivatives of such mercapto compounds as dodecyl mercaptan, oleyl mercaptan, cetyl mercaptan, decyl mercaptan and thiophenols, thionaphthols, and benzylmercaptan; also the polyalkylene oxide derivatives, such as those described in U.S. Patent No. 2,085,706, of carboxylic acid amides, and sulfonamides of the type described in U.S. Patent No. 2,002,613, or the polyalkylene oxide derivatives, described in U.S. Patent No. 2,266,141, of sulfonic acids may be employed if desired.

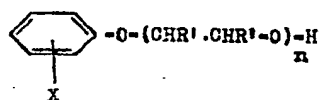
What we claim is:—

1. A germicidal detergent composition comprising a solution of a germicidally effective amount of free iodine in a water soluble, nonionic surface active agent containing a polyglycol ether group and having the general formula:



wherein R represents a residue of a water insoluble organic compound containing at least 6 carbon atoms and having an active hydrogen atom. R^1 represents hydrogen or lower alkyl and n represents an integer of from 3—100 or higher.

2. A composition as defined in claim 1 wherein the nonionic surface active agent specified is a polyglycol ether of an alkyl phenol containing 6 to 18 alkyl carbon atoms, said nonionic surface active agent having the formula:



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